Process Optimization for Laser Gas Nitriding of Shape Memory NiTi alloys

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Introduction

Near-equiatomic nickel-titanium (NiTi) alloy, which is well known for its unique shape memory and superelastic properties, have been widely used in various biomedical applications, such as cardiology, vascular stents, staple and knee joints [1-2]. However, from the perspective as a bio-metallic material, the relatively inferior wear resistance of NiTi is a big concern as it will increase the chance of releasing toxic Ni ion when the surface is worn-off in service [3-4]. Surface modification is therefore required to improve the wear resistance of NiTi.

One common approach to improve the wear resistance is to increase the surface hardness, i.e. the higher the hardness, the higher the wear resistance. Laser gas nitriding (LGN) was used in this study to increase the surface hardness of NiTi, given that it has the benefits of high efficiency, ease of control and automation, and high precision for the treatment location [5].

Experimental Details

Laser Gas Nitriding (LGN) process was performed using a 100W CW fiber laser (SP-100C-0013, SPI and A&P Co., Ltd) with a wavelength of 1091nm. The samples were carried out in a specially-designed gas chamber which was continuously disgorged with pure nitrogen gas at a rate of 20 L/min. A series of experimental runs were conducted to determine the optimal set of processing parameters: laser power, scanning velocity and beam diameter to obtain the highest surface hardness. Table 1 shows the range of parameters varied in the LGN experiments:

Table 1. Choice of processing parameters

Factor	Range of parameter
Output power	70 W – 90 W
Scanning velocity	300 mm/min - 900 mm/min
Beam diameter	0.4 mm – 0.6 mm

Results and Discussion

From the results of LGN experiments, the optimal parameter set was found to be 90 W output power, 300 mm/min scanning velocity, 0.4 mm laser beam diameter and 20 L/min nitrogen gas flow rate. The surface hardness of NiTi after LGN was determined by Vickers micro-hardness test provided that the thickness of TiN deposited on the surface was thick enough to avoid the substrate effect. The Vickers hardness method consists of indenting the test material with a diamond indenter, which is to form a right pyramid with a square base with 136^{o} between opposite faces, the schematic diagram of Vickers hardness test is depicted in Figure 1.

Figure 1. Schematic diagram of the Vickers hardness test method

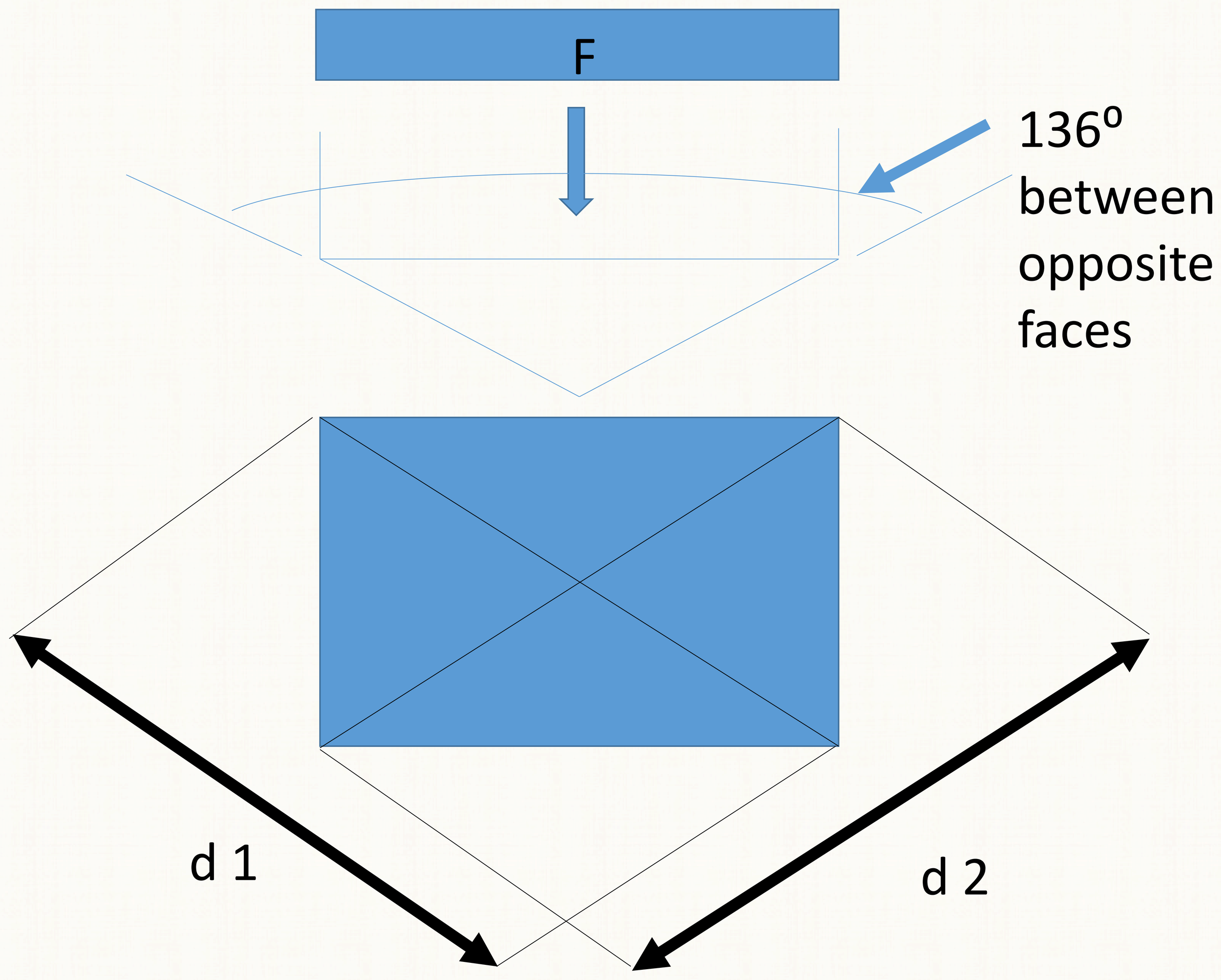

Table 2. Vickers hardness values of laser treated surface and bare NiTi

	Vickers Hardness (HV)
Laser treated surface (TiN)	700 HV ± 68
Bare NiTi	360 HV ± 40

The Vickers hardness results in Table 2 shows that the hardness of the laser-nitrided surface reached about 700 HV ± 68 which was nearly twice than that of the bare NiTi. The increased hardness was due to the formation of the very hard nitride layer by LGN. The nitride formation mechanism can be represented by :

$$Ti \rightarrow \alpha\text{-}Ti\,(N) \rightarrow Ti_2N \rightarrow TiN$$

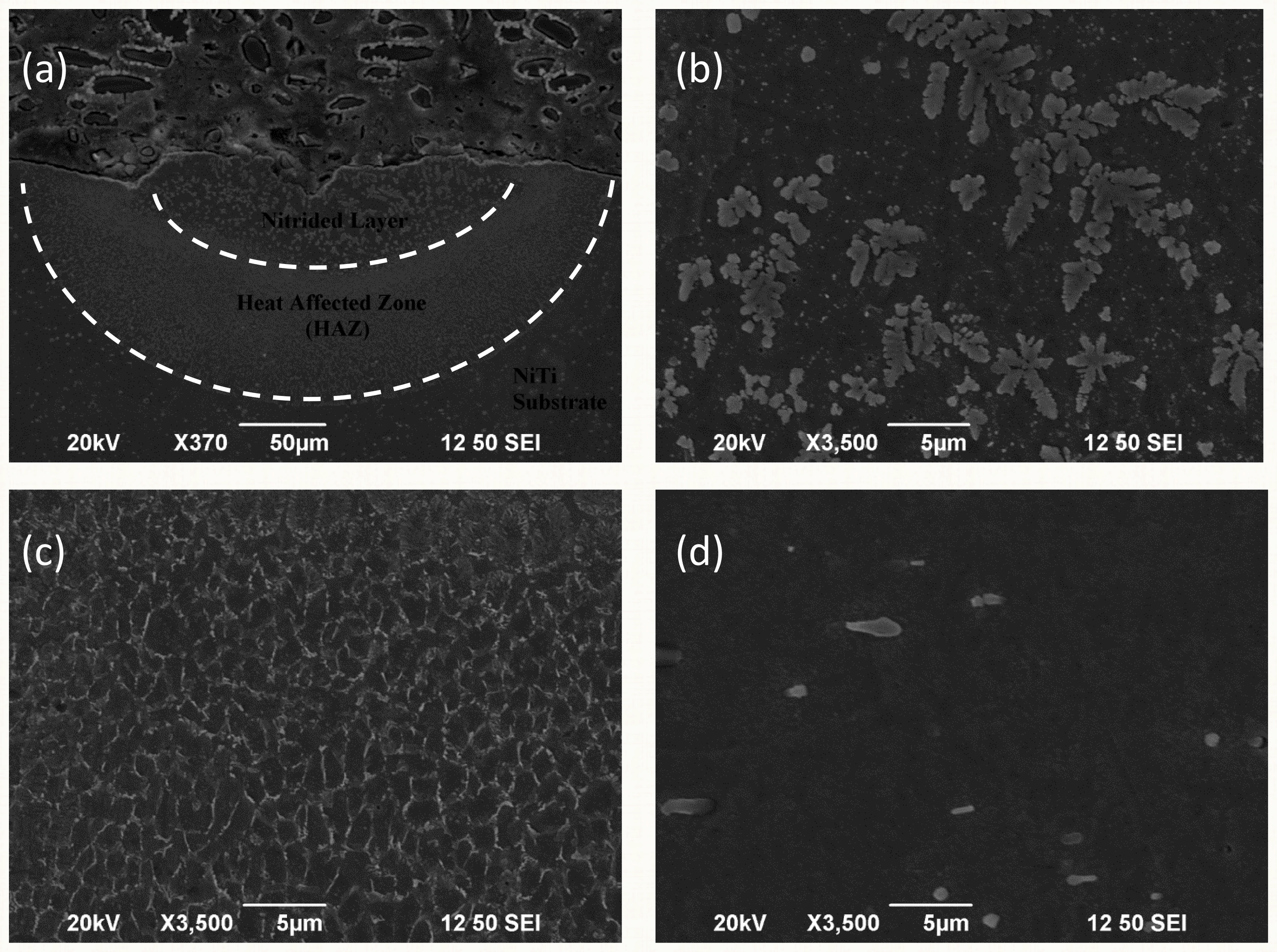

Figure 2. Microstructure of the cross-section view of laser gas nitrided NiTi (a) all three zones, (b) laser gas nitrided layer, (c) heat affected zone, and (d) NiTi substrate

The SEM micrographs in Figure 2 show the microstructure of the laser-nitrided sample. Three regions could be identified, namely, the nitrided zone (or TiN), the heat affected zone (HAZ), and the NiTi substrate. There are marked differences among all three regions in Figure 2(a). The TiN layer had a thickness of 50 µm to 60 µm. After etching, a dendritic phase of TiN was clearly visible (Figure 2(b)). The microstructure was fairly homogeneous in the HAZ as depicted in Figure 1(c).

Figure 3 shows the XRD patterns for the laser-nitrided NiTi and bare NiTi. Strong peaks corresponding to TiN (111), (220) and (311) were present in the pattern for the laser-nitrided sample, confirming the formation of TiN after LGN.

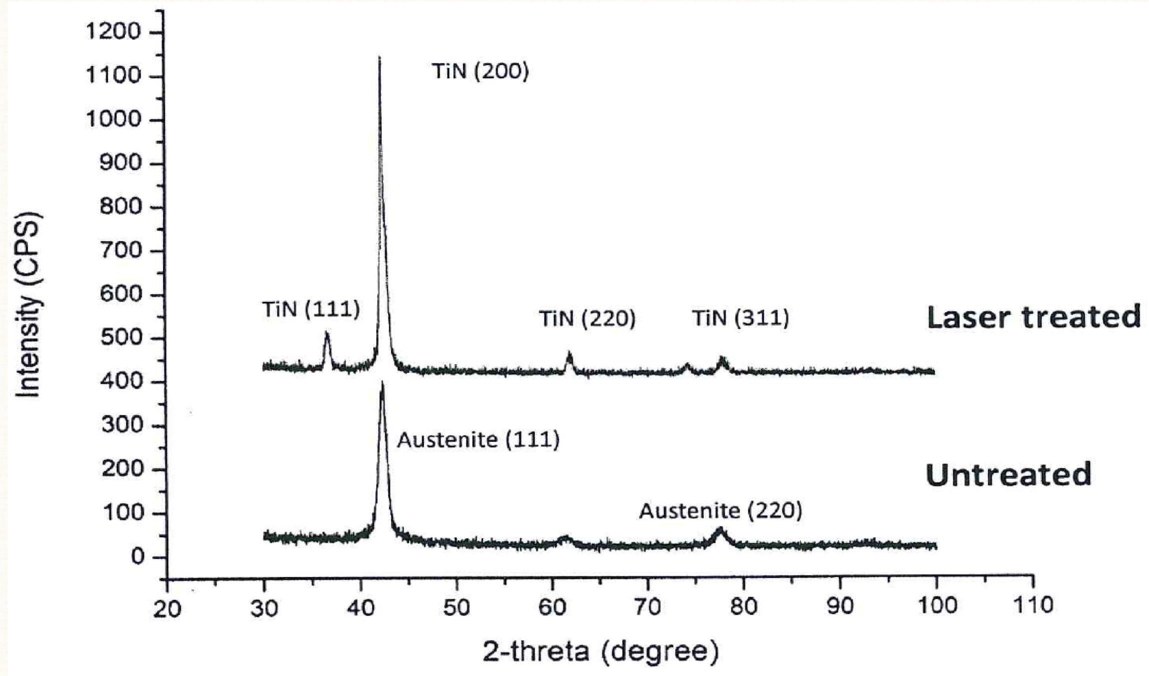

Figure 3. XRD patterns for laser treated and untreated sample

Conclusions

In this study, an optimal set of laser parameters to deposit a TiN layer on NiTi were identified. The microstructural and mechanical properties of the layer were determined as follows:

1. The optimum parameter combination were 90 W (laser output power), 300 mm/min (laser scanning velocity), 0.4 mm (laser beam diameter) and 20 L/min (nitrogen gas flow rate).
2. A layer of TiN was formed on the surface of NiTi with the thickness about 50 µm to 60 µm. A dendritic phase could be observed in the nitrided zone. The Vickers hardness of the TiN was about twice than that of the bare NiTi.

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